

# Observation of Quartz Cathode-Luminescence in a Low Pressure Plasma Discharge

John E. Foster  
Glenn Research Center, Cleveland, Ohio

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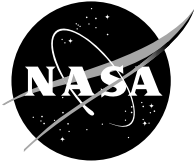
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# **Observation of Quartz Cathode-Luminescence in a Low Pressure Plasma Discharge**

John E. Foster  
National Aeronautics and Space Administration  
Glenn Research Center  
Cleveland, Ohio 44135

## **Summary**

Intense, steady-state cathode-luminescence has been observed from exposure of quartz powder to a low pressure rf-excited argon plasma discharge. The emission spectra (400 to 850 nm) associated with the powder luminescence were documented as a function of bias voltage using a spectrometer. The emission was broad-band, essentially washing out the line spectra features of the argon plasma discharge.

## **Introduction**

Cathode luminescence of non-conductive materials such as quartz and alumina has been documented in the past. Cathode luminescence in general is the emission of light from a solid caused by the impact of energetic electrons. The electron impacts with the surface can give rise to surface electronic excitation.<sup>1</sup> Subsequent relaxation results in the release of photons (luminescence). If such impacts are energetic enough, dislocations in the lattice of the solid can be generated. Such dislocations can be subsequently occupied by an electron.<sup>2</sup> These so-called F-centers can then be excited via absorption of energy. De-excitation leads to the emission of photons and thus the resulting luminescence. Peters observed fluorescence of quartz under the influence of cathode rays.<sup>3</sup> In this case, cathode ray collisions with quartz discharge tube walls generated a red fluorescence. Saksena and Pant observed feeble quartz luminescence upon exposure of the quartz to a 70 000 eV electron beam.<sup>4</sup> The luminescence was attributed to the formation of F-centers in the quartz, presumably, formed via modification to the Si-O group in the crystal. F-center derived luminescence has also been observed in various forms of alumina and in particular in those applications where alumina is used for high power rf windows.<sup>5,6,7,8,9</sup> Intense alumina emission has been observed as a consequence of collisions of energetic electrons formed during multipactor processes with the alumina surface.<sup>7-10</sup>

In general, intense, lower energy (<1 kV) luminescence is of general interest in that utilization of such phenomena for spectroscopy or lighting can be readily implemented. Cathode luminescence of materials such as silicon dioxide at energies as low as 200 V has also been documented.<sup>11</sup> In the work presented here, very intense luminescence of quartz and alumina powders has been observed upon exposure to electron flux extracted from a low pressure rf discharge plasma. The observed luminescence in the visible occurred at electron energies between 400 and 1000 eV. This report documents the acquired emission spectra, discharge conditions, as well as electron current flux to the powder holder as a function bias voltage.

## **Experimental Set-up**

Quartz and alumina powder luminescence was studied in a 30 cm diameter multipole plasma source. The plasma source discharge was sustained using a 1.5 turn internal, copper antenna. The

antenna was excited at 13.56 MHz using an rf power supply and matching network. For this work, the discharge power (rf) was varied between 10 W and 150 W. In all cases, the reflected power was less than 2 W. The source itself contained 3 quartz windows, as illustrated in figure 1a. The windows allowed for the examination of dusty plasma phenomena<sup>12</sup> and powder emission, which took place at the dust holding electrode. A schematic representation of the source with the dust holding electrode is shown in figure 1b. The dust holding electrode was located between magnetic cusps at the wall of the plasma source. As further detailed in figure 2, the dust holding electrode contained two rows of samarium cobalt permanent magnets of alternating polarity located behind the stainless steel dust holding electrode. The magnets effectively coupled the dust holder into the plasma source magnetic circuit. It was found that the most intense cathode-luminescence was observed along the line of permanent magnets in the dust holder. The dust holding electrode could be biased up to +1 kV relative to ground.

Argon gas was introduced into the source via a multi-orifice gas plenum ring. The exit plane of the source contained a grid, which allowed for the evacuation of the source. The source itself was tested in a 1-m diameter by 2.2-m long cylindrical vacuum facility evacuated by a 30 cm turbo-pump. The pumping speed of the chamber was approximately 2000 liters/s. Typical base pressure was  $2 \times 10^{-5}$  Torr. During discharge operation (gas flow), the chamber pressure varied between  $8 \times 10^{-5}$  Torr and  $1.5 \times 10^{-4}$  Torr.

A double Langmuir probe was used to estimate plasma properties of the discharge as a function of rf power. The double probe is a floating probe and is not as sensitive to rf plasma oscillations imposed by the rf antenna.<sup>13</sup> The double probe's collection elements consisted of two 5 mm long, 0.38 diameter tungsten tips. The probe was mounted such that the probe extended 10 cm into the discharge chamber and 3 cm downstream of the antenna to sample the core plasma.

Plasma induced emission could be observed through three orthogonal quartz windows located on the discharge chamber. The light from the plasma was studied using a ccd camera which was trained on the discharge chamber windows through the vacuum chamber view-port. The plasma-induced light emission and the powder luminescence were also collected by an optical fiber located at a window just downstream of the biasable electrode on which the quartz or alumina dust was placed. The fiber coupled the collected light into a spectrometer. The spectrometer used in this investigation had a resolution of approximately 0.5 nm.

The sub-micron to micron sized dust particles used in this investigation were obtained from a commercial source. Though cathode-luminescence was observed both for quartz and alumina powders, the bulk of this work focuses on observations and measurements made with quartz particles of average diameter 45 microns. These particles were placed uniformly over the surface of the dust holding electrode before each test.

## Experimental Results

Cathode luminescence of the quartz dust was observed when the powder-holding electrode was exposed to the argon plasma and then biased positively relative to ground potential. Presumably, energetic electrons bombard the dust on their way to the electrodes, thereby giving rise to surface excitation. In all cases investigated, the most pronounced surface luminescence was observed to occur at those regions located above the dust holder magnets. This is likely attributed to the fact that electron collection occurs primarily at the center of the magnetic cusps. As such, the electron current density at the cusps is considerably higher than what it would be if it were collected uniformly over the surface of the entire electrode. The concentration of electron flux both localizes and intensifies the resulting emission. The electron current collected at the magnetic cusps is a function of the plasma density, and by increasing discharge power, the current to the magnetic cusps can be increased. The plasma sheath electric field at the dust surface and the presence of space charge neutralizing ions also improve

current throughput at the cusps by relaxing space charge limitations. Figure 3 illustrates variations in cathode luminescence as a function of bias voltage at approximately fixed rf power, as acquired using a ccd camera trained on the 45 micron quartz powder. The magnets were present in the dust holder for the conditions photographed in the figure. Figure 3b shows that at low bias voltages, a visible, bluish glow at the dust surface could be observed. This bluish luminescence, which is observable in figure 3b and 3c (at 25 and 50 mA, respectively) heralded the onset of the subsequent intense glow at the magnetic cusps. Coverage of the bright glow over the surface of the quartz powder increased with increasing bias voltage as shown in figures 3d and 3e.

As can be seen in figures 3a to 3e, the cathode luminescence intensity increases as the bias voltage is increased from 0 to 600 V. The bias current to the electrode also increased with increasing bias voltage. Figure 4 illustrates typical variations in the dust electrode current as a function of the dust holding electrode bias voltage. As shown in figure 4, the current to the holder increases with increasing bias voltage, with a significant change in slope just below 500 V. Above 500 V, the current increases at a much larger rate (6×) with increasing voltage. This jump occurs for both input rf power levels shown: 50 and 85 W. The dust luminescence is also most intense at voltages above this value. This observation is consistent with the expected increase in cathode-luminescent events with increasing electron current and electron energy (bias voltage). The exact nature of the rather dramatic increase in slope is likely due to the onset of increased electron production: (1) local electron bombardment ionization and (2) secondary electron emission. Secondary electron emission of quartz has a maximum between 400 and 440 eV.<sup>14</sup> It is likely that of the two mechanisms mentioned, secondary emission may be dominant at these energies. These additional electrons contribute to the total electron current that impacts the quartz dust and are ultimately collected at the electrode below.

The bright glow from the dust particles eventually overwhelms the discharge glow, thereby dominating the total emission spectra as the bias voltage was increased. Figure 5 depicts variation in the measured emission spectra as a function of bias voltage. Without a bias voltage (figure 5a), the emission spectra are characterized as line spectra typical of a low pressure argon plasma discharge. As the bias voltage increases, however, the baseline is distorted (figure 5b). Eventually the spectral profile associated with the dust emission dominates with line spectra features superimposed (figure 5c). The broad-band nature of the cathode luminescent signal is similar in form to that observed in other systems.<sup>7</sup> It should be pointed out that the location of the peak (around 600 nm) in the observed broad profile is consistent with silicon dioxide cathode luminescent spectra profiles recorded by other researchers.<sup>15,16</sup>

The high intensity of the glowing dust suggested that electron heating of the quartz could also be contributing to the observed spectra. If this contribution dominates, then it might be expected that the emission profile should take on a blackbody-like intensity characteristics. According to Wien's law, if the emission spectra were purely attributed to black body radiation, then the characteristic temperature of the quartz would be approximately 4800 K. Quartz is molten at 600 °C. Quartz and alumina dust investigated here would vaporize well before reaching such high temperatures. Additionally, continuum radiation, which is typically observed in high pressure (in contrast to the low pressure conditions investigated in this work) can also be ruled out as a contributor to the emission profile.<sup>17,18</sup> In this regard, it is unlikely that intense dust heating or continuum radiation were the dominant contributors to the spectral profile, leaving electron-induced luminescence as the primary mechanism. Additionally, post-test visual observations revealed the surface of the dust discolored with a brownish tint suggesting possible F-center formation.<sup>19</sup> It is noteworthy that the observed cathode-luminescence emission provides a profile that could otherwise only be achieved from a very hot blackbody at an emission temperature considerably higher than conventional material melting points.

On average, luminescence tests at a given plasma condition were operated up to 0.5 hr. It should be pointed out that upon post-test removal of the quartz powder from the dust holding electrode, the surface of the electrode was observed to be relatively unchanged, with no evidence of melting as would be otherwise associated with an arcing event or from significant electron heating.

The fact that the distribution of emitted spectra is so broad suggests that this luminescent-phenomena could be exploited as a potential broad band light source without the requirement of a very hot source or a very high pressure discharge. The phenomena observed here emit broad band radiation in the visible, ranging from roughly 400 nm to over 850 nm. A light source with such a profile could be used in absorption spectroscopy applications. Because the band extends over the visible range, a source exploiting this process may also be applicable to solid-state lighting applications.

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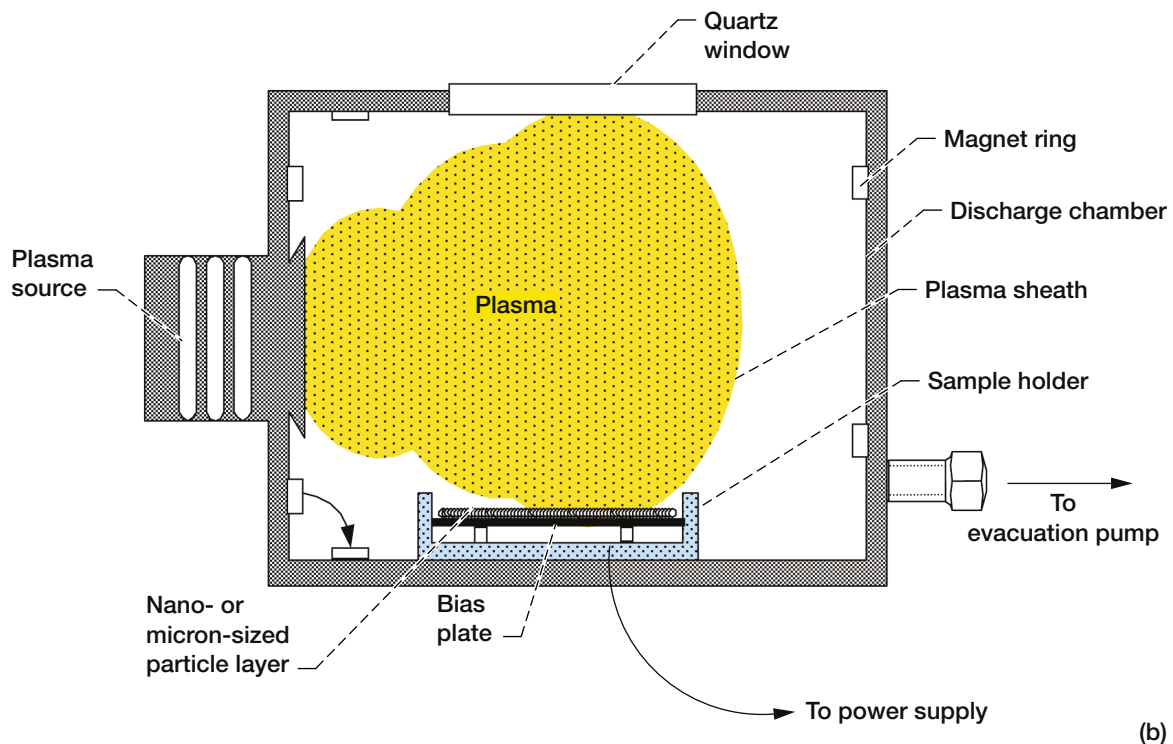
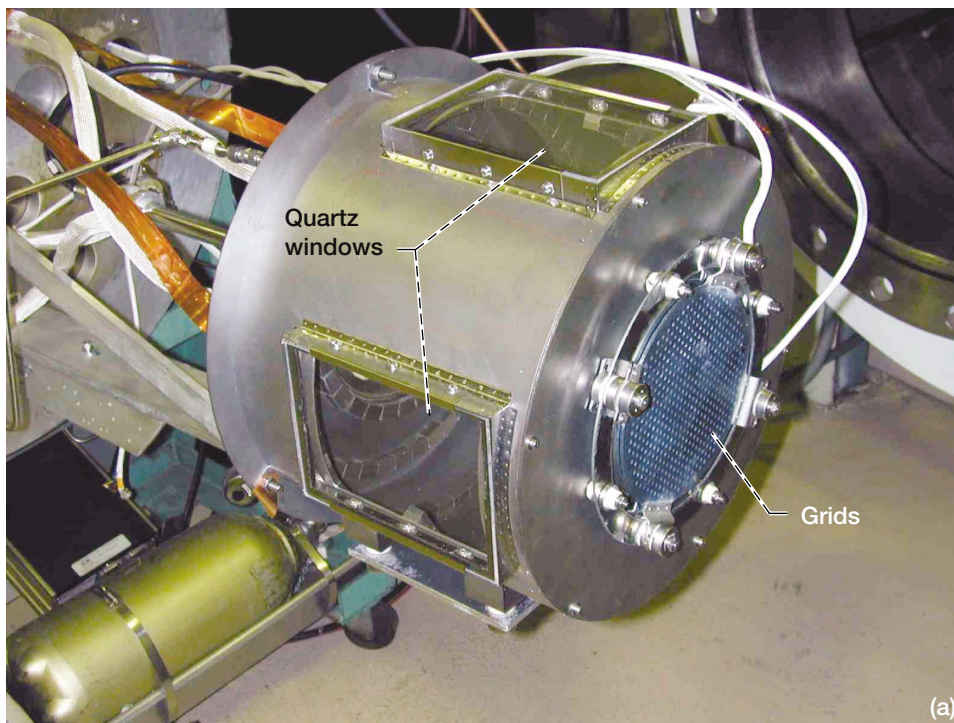


Figure 1.—Photo of plasma source and schematic of plasma. (a) Photograph of the plasma source in which catho-luminescence was studied. Notice quartz windows used to observe light emission. (b) Schematic depiction of the plasma generated by the plasma source and the dust holding electrode.

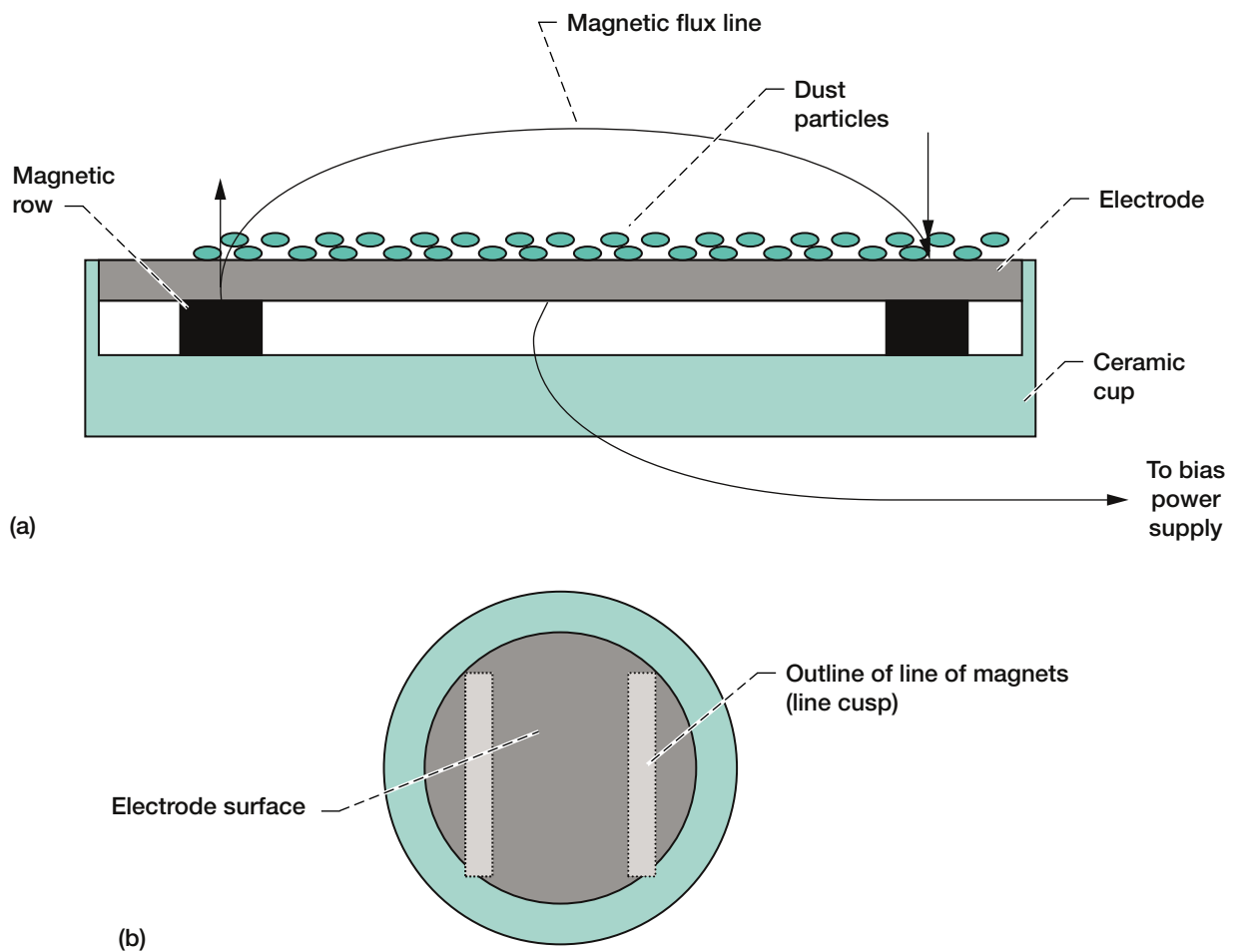


Figure 2.—Schematic depiction of the dust holding electrode. The most intense luminescence was observed to occur at the magnetic cusps. (a) Side view. (b) Top view.

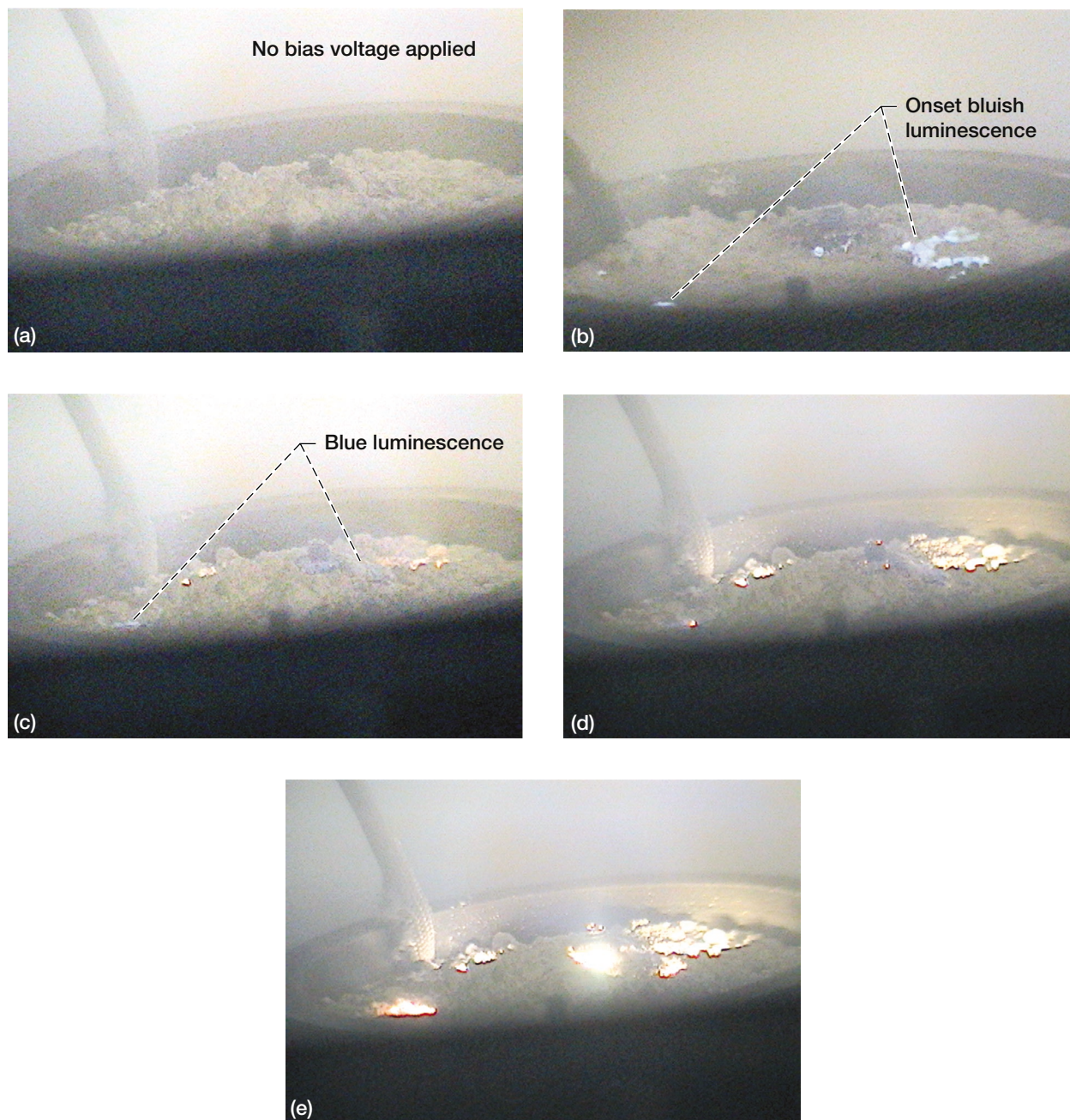


Figure 3.—Observed luminescence as a function of dust electron current. (a) Current = 0 (floating). (b) Collected current = 25 mA, 400 V. (c) Collected current = 50 mA, 500 V. (d) Collected current = 70 mA, 520 V. (e) Collected current = 100 mA, 560 V.

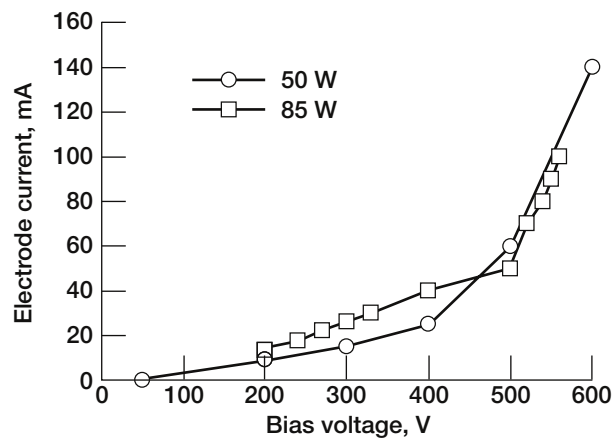


Figure 4.—Variation in dust electrode current with increasing bias voltage. Note the change in slope that occurs near 500 V.

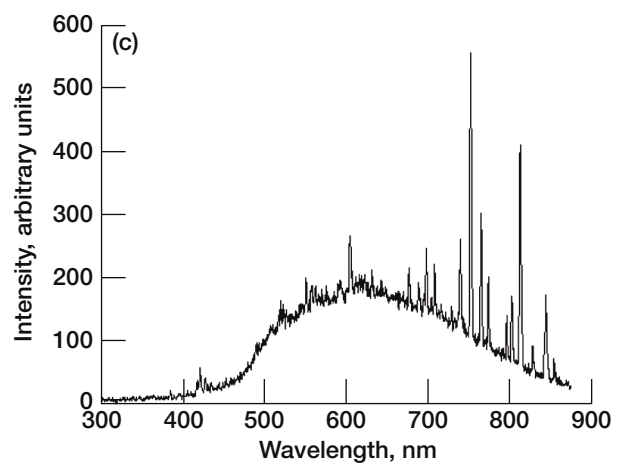
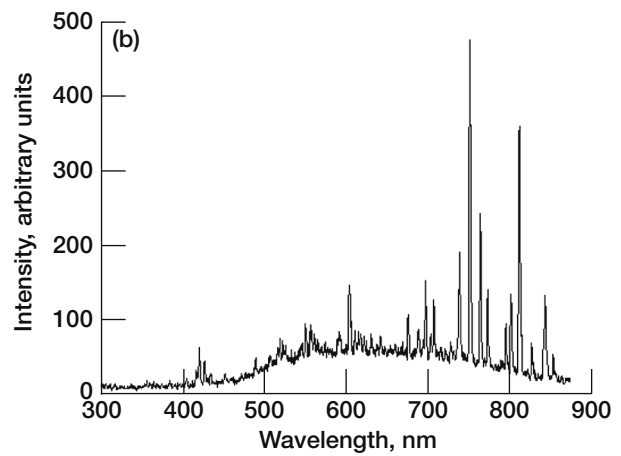
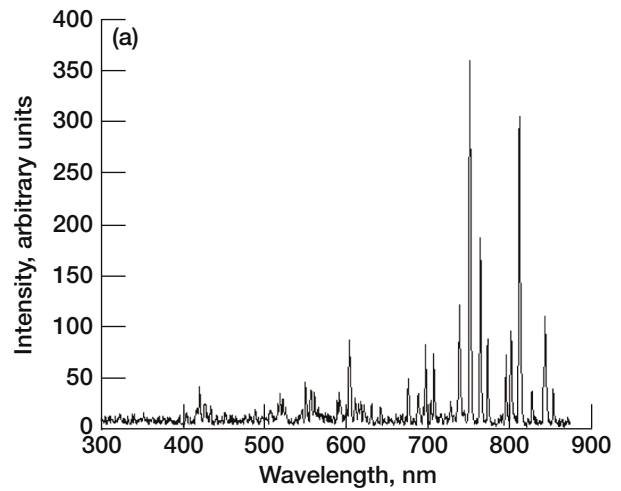


Figure 5.—Variation in the observed emission spectra as a function of bias voltage. (a) 0 V. (b) 580 V. (c) 660 V.



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